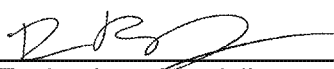
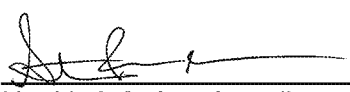
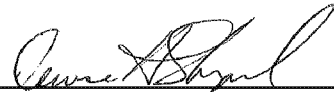



Title: SUBSAMPLING

[Method: ASTM D6323-12]

Approvals (Signature/Date):

 Technology Specialist	12/03/14 Date	 Health & Safety Coordinator	12/08/14 Date
 Quality Assurance Manager	01/22/15 Date	 Technical Director	01/15/15 Date

This SOP was previously identified as SOP No. NC-OP-046, Rev 0, dated 11/27/14

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1. SCOPE AND APPLICATION

- 1.1. These mixing, subsampling, and splitting techniques are applicable to a wide range of soil, sediment, tissue, water, and waste samples. Care must be taken to match the appropriate technique with the matrix, target analytes and quality objectives.
- 1.2. The goal of all mixing, subsampling, and splitting techniques is to obtain representative splits or subsamples for preparation and analysis. Particle size reduction and processing can be beneficial in obtaining representative subsamples and specific processes for particle size reduction and processing can be found in the soil processing SOP (NC-OP-044) and non-soil processing SOP (NC-OP-045).
- 1.3. This document accurately reflects current laboratory Standard Operating Procedures (SOP) as of the date above. All facility SOPs are maintained and updated as necessary.
- 1.4. **NOTE:** If foreign or quarantined solids are received, refer to SOP NC-SM-019 Canton Foreign Soils, current revision, and contact your Environmental Health and Safety Coordinator for proper handling instructions.

2. SUMMARY OF METHOD

- 2.1. All samples should be mixed and subsampled appropriately for the test and analytes of interest.
- 2.2. Solid sample mixing procedures include in-jar mixing, horizontal surface mixing, and mortar and pestle.
- 2.3. Solid sample subsampling procedures include alternate scoop, one-dimensional slabcake, two-dimensional slabcake, and cone and quarter methods.
- 2.4. Liquid sample subsampling procedures include centrifuge, pipettes, coliwasa devices, and multiphase procedures.

3. DEFINITIONS

- 3.1. Refer to the glossary in the TestAmerica Canton Quality Assurance Manual (QAM), current version.
- 3.2. Mix: To thoroughly blend the sample and reduce the analyte concentration differences between different parts of the overall sample. It is most effective when the particle size and density differences within the sample are small.
- 3.3. Incremental Sampling Methodology: A structured composite sampling and processing protocol that reduces data variability and provides a reasonably unbiased estimate of the

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mean contaminant concentrations in an area/volume of soil targeted for sampling. ISM provides representative samples of specific soil areas/volumes defined as decision units (DUs) by collecting numerous increments of soil (typically 30-100 increments) that are combined, processed, and subsampled according to specific protocols.

- 3.4. Grinding: A generic term for soil disaggregation or milling.
- 3.5. Disaggregation: The act of breaking the soil clumps into individual small particles but keeping the small pebbles and hard crystalline particles intact.
- 3.6. Milling: Complete particle size reduction of all soil components including hard crystalline materials to a defined maximum particle size (e.g. <250 µm or <75 µm).
- 3.7. Sample: For laboratory technicians, the sample is all the material delivered to the laboratory in a container collected by the field crew.
- 3.8. Subsample: The small representative amount removed from a field sample selected for final analysis. This is also referred to in some SOPs as the aliquot.
- 3.9. Representative subsample: A subsample taken in such a way that each particle had an equal chance of being selected. The most representative subsample is one that most closely resembles the true value of the material sampled or contains the least amount of error.

4. INTERFERENCES

- 4.1. Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts. All of these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section of each analytical SOP. Specific selection of reagents may be required to avoid introduction of contaminants.
- 4.2. Metallic components of particle size reduction equipment can contribute some metal content to the solid samples. Hence carbon steel components are usually preferable to stainless steel when necessary to minimize contamination from chromium, nickel and molybdenum. Some contamination from iron is common.

5. SAFETY

- 5.1. Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual, the Facility Addendum to the Corporate EH&S Manual, and this document.
- 5.2. Eye protection that protects against splash, laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Cut-resistant gloves must be worn doing any other task that presents a strong possibility of

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getting cut. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately.

- 5.3. Exposure to chemicals must be maintained **as low as reasonably achievable**; therefore, unless they are known to be non-hazardous, all samples must be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation where possible. All samples with stickers that read "Caution/Use Hood!" **must** be opened in the hood. Contact the EH&S Coordinator if this is not possible. Solvent and waste containers will be kept closed unless transfers are being made.
- 5.4. The following is a list of the materials used in this method, which have a serious or significant hazard rating. NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the Safety Data Sheet (SDS) for each of the materials listed in the table. A complete list of materials used in the method can be found in the Reagents and Standards section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Methanol	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Overexposure may include headache, drowsiness and dizziness. Methanol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur: symptoms may be parallel to inhalation exposure. Irritant to the eyes.
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

- 5.5. All work must be stopped in the event of a known or potential compromise to the health and safety of a TestAmerica associate. The situation must be reported **immediately** to the EH&S Coordinator and the Laboratory Supervisor.
- 5.6. When operating the electric chopper or grinder, be sure to keep all aqueous liquids clear to prevent the risk of electrical shock from any spills.
- 5.7. Avoid inhalation of sample dust. Work in a ventilation hood when necessary to avoid accidental dust inhalation. Wear a dust mask or respirator if the ventilation hood does not provide sufficient dust protection.

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- 5.8. If there is any malfunction in the equipment. De-energize and tag out.
- 5.9. All noise levels are below OSHA limits.
- 5.10. Only trained personnel are permitted to use crushing equipment. A list of trained personnel will be maintained with EH&S and QA.

6. EQUIPMENT AND SUPPLIES

- 6.1. Butcher Paper
- 6.2. Plastic wrap
- 6.3. Aluminum foil
- 6.4. 8 oz. Glass jars with lids
- 6.5. Top-loading balance.
- 6.6. Appropriate containers for each subsample

7. REAGENTS AND STANDARDS

- 7.1. Deionized water: Reagent water must be produced by a Millipore DI system or equivalent. Reagent water must be free of the analytes of interest as demonstrated through the analysis of preparation blanks.
- 7.2. Methanol: VOC grade
- 7.3. Acetone: Pesticide grade

8. SAMPLE COLLECTION, PRESERVATION, AND STORAGE

- 8.1. Not applicable to this procedure. Sample collection, preservation, and storage are dependent on the requested test method and analytes.

9. QUALITY CONTROL

- 9.1. Equipment processing blanks might be applicable for some particle size reduction or selection techniques. There is no single blank matrix that is suitable for all analytes, equipment or processes. The blank matrix should be chosen by the client/data user based on the advantages and limitations described below in Section 9. If no blank matrix is selected by the client, reagent water should be used as the default when possible, as noted in Section 9.2.

- 9.2. Reagent water (or organic solvents) may be used to rinse the equipment surfaces. This liquid is then analyzed for the target analytes. This process is good at monitoring residue on equipment surfaces from previously processed samples. It does not evaluate the potential contribution of the equipment surface material to a solid sample.
- 9.3. Sand may be run through processing equipment and then analyzed to monitor for both sample carryover residue and contamination from equipment surface erosion. This process is most applicable for organic analytes. Sand always contains metals. These metals concentrations might be too high for suitable blank demonstrations. Also, the sand material is frequently more abrasive than soil and can over estimate sample contamination due to erosion of the equipment surfaces.
- 9.4. Teflon boiling chips can be suitable to monitor the cleanliness of processing equipment surfaces. This option is between reagent water and sand regarding abrasion of the equipment surfaces. This material is generally non-detect at parts per billion concentrations for most organic and inorganic analytes. The Teflon material does not mimic the behavior of soil in the subsequent sample extraction or digestion procedures.
- 9.5. Reprocess half of a field sample and look for differing analyte concentrations between the single and double processed sample aliquots. The occurrence of significantly elevated analyte concentrations in the double processed sample can be a good indication of carryover or contamination due to equipment surface erosion. This is most applicable for monitoring metals contamination in soil grinders.

10. CALIBRATION AND STANDARDIZATION

- 10.1. Not applicable to this procedure.

11. PROCEDURE

- 11.1. One-time procedural variations are allowed only if deemed necessary in the professional judgment of QA, operations supervisor, or designee to accommodate variation in sample matrix, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo.
- 11.2. Any unauthorized deviations from this procedure must also be documented as a nonconformance with a cause and corrective action described.
- 11.3. Sample Preparation
 - 11.3.1. The following sections describe a variety of procedures. Additional procedures for sample preparation are available in the Soil and Non-Soil Processing SOPs. The client/data user must select the procedure that is most appropriate for the sample matrix, analytes, and quality objectives. The selection of the procedure(s) can be made by the client in consultation with the appropriate TestAmerica representatives during project planning. There is no single default procedure that can be used in the absence of client selection.

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11.3.2. Solid Sample Mixing Procedures

11.3.2.1. In jar mixing

11.3.2.1.1. The flowable sample is thoroughly stirred in the sample jar using a wooden blade.

11.3.2.2. Horizontal surface mixing

11.3.2.2.1. The sample is transferred to an aluminum tray or onto a sheet of paper and mixed with a wooden blade.

11.3.3. Solid Sample Subsampling Procedures

11.3.3.1. Multiple increments from a jar

11.3.3.1.1. Select 5-10 small sample increments from various locations within the sample container to make the entire subaliquot. The increments must come from all general areas of the container-top, sides, center, and bottom.

11.3.3.2. Two dimensional slabcake

11.3.3.2.1. Spread the sample to a consistent depth on a clean flat area covered with an appropriate disposable cover (butcher paper, aluminum tray or foil or plastic depending on the analytes of interest).

11.3.3.2.2. Select 30 or more small increments spread evenly over the sample area or as dictated by client data quality objectives. The increments must be collected with a blunt end spatula or scoop to evenly collect from the top middle and bottom of the slabcake. A coring device of suitable material can also be used if the sample is sufficiently cohesive (e.g. moist soil).

11.3.3.2.3. If the sample has noxious odors or produces dust, the sample spreading and increment collecting should be performed in a hood or large flat bag of appropriate material. The analyst must be protected from potential inhalation hazards from the sample.

11.3.3.3. Alternate scoop

11.3.3.3.1. Wet or dry solid samples can be divided into two or more smaller aliquots. Determine the number of sub-aliquots, and prepare that many empty containers.

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- 11.3.3.3.2. Scoops of the mixed laboratory sample are either placed in the analytical vessel or discarded.
- 11.3.3.3.3. Three scoops are discarded for every scoop saved. Randomly select an aliquot and place in the first container. Place the next three aliquots in the second container. Repeat as needed for additional aliquot containers.
- 11.3.3.3.4. Repeat the aliquoting cycle until the original sample is consumed.
- 11.3.3.3.5. The alternate scoop technique can also be used to subaliquot discrete samples that are to be homogenized into one composite sample.
 - 11.3.3.3.5.1. Determine approximately the ratio of aliquot to discard scoops required to consume the sample and achieve the aliquot size required for homogenization. For example, if a 50 g aliquot is desired from a 250 g sample, a ratio of one scoop used to four scoops discarded will be used.
 - 11.3.3.3.5.2. For each discrete sample, set up two empty containers of appropriate volume.
 - 11.3.3.3.5.3. Stir each discrete sample in its container, or if necessary homogenize on a sheet of butcher paper and return to the jar.
 - 11.3.3.3.5.4. Place one of the empty containers on a balance and tare it. Place one scoop of sample from the top of the discrete sample jar into this container. This will be the portion used for a homogenized composite later.
 - 11.3.3.3.5.5. Into the second jar, place the three or more (as determined in Section 11.3.3.3.5.1) scoops to be discarded.
 - 11.3.3.3.5.6. Repeat Sections 11.3.3.3.5.4 and 11.3.3.3.5.5 until the sample has been consumed and the desired weight of the subaliquot is in the tared container. Record the weight of the sub-aliquot.
 - 11.3.3.3.5.7. Repeat this process for all of the discrete samples that will be used for the composite, using

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approximately equal weights of each discrete sample.

- 11.3.3.3.5.8. After all discrete samples have been sub-aliquoted using the alternate scoop procedure, combine them on a sheet of butcher paper and homogenize. Pour the homogenized sample volume into the labeled sample jar for the composite sample.

11.3.3.4. One dimensional slabcake

- 11.3.3.4.1. This process is intended to produce large sub-samples from very large dry flowable solid samples, such as those collected using incremental sampling methodology, dried and then chopped using the process described in Section 1.1.1See Reference 16.1.7.
- 11.3.3.4.2. Pour the dry soil sample into a long thin pile onto a clean horizontal surface. The pour height should not exceed 20 cm to minimize the formation of a dust cloud.
- 11.3.3.4.3. Ensure that the sample container makes at least 20 passes back and forth over the "line of sample".
- 11.3.3.4.4. Using a rectangular flat-bottomed scoop, remove an increment from the "line of sample". Ensure that a complete cross cut of the sample line includes the entire depth of that increment. Combine increments as needed to produce the needed sub-sample size.

11.3.3.5. Cone and quarter (for dry flowable samples)

- 11.3.3.5.1. Pour the sample into a cone on a clean flat area covered with an appropriate disposable cover (butcher paper, aluminum foil, or plastic depending on the analytes of interest).
- 11.3.3.5.2. Cut the cone in half in two directions to form four quadrants.
- 11.3.3.5.3. Return opposite quadrants to the original sample container.
- 11.3.3.5.4. Repeat the process in Sections 11.3.3.5.1 through 11.3.3.5.3 until the needed aliquot size has been obtained.

11.3.4. Liquid Sample Mixing Procedures

11.3.4.1. Closed container shaking

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11.3.4.1.1. All liquid samples should be mixed by shaking in the original closed sample container unless a multiple layer sub-aliquoting procedure is used. This applies to emulsifiable layers such as oil and water and suspendable particulates in water. The sample must remain mixed long enough to pour out a representative aliquot. Samples that show noticeable separation in under a minute should be sub-aliquoted using an appropriate technique as described below.

11.3.4.1.2. The shaking process must be vigorous enough to mix and distribute analytes associated with different layers, particulates, or inside container walls.

11.3.5. Liquid Sample Subsampling Procedures

11.3.5.1. Pour

11.3.5.1.1. Samples that are well mixed can be sub-aliquoted by pouring an appropriate volume off the top of the sample.

11.3.5.2. Layer Subsampling

11.3.5.2.1. Some liquid samples with multiple layers separate too quickly to pour a representative sub-aliquot off the top. In some instances it is best to separate the layers and handle them as individual samples. In other instances representative aliquots of each layer must be collected and processed as a single sub-sample.

11.3.5.2.2. Unless directed otherwise, record the phase ratios (or volumes) of the layers.

11.3.5.2.3. Gravity or Centrifuge Settling

11.3.5.2.3.1. Allow the sample layers to separate based on density. Centrifugation can be used to accelerate the process if simple gravity settling is not fast enough.

11.3.5.2.4. Separatory Funnel

11.3.5.2.4.1. Gently pour the sample into a separatory funnel. Allow time for additional layer separation as needed.

11.3.5.2.4.2. Drain the layers out into separate containers one at a time.

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- 11.3.5.2.4.3. Some oils stick to the separatory funnel sides so the draining process must be slow enough to avoid remixing the layers.

11.3.5.2.5. Pipette

- 11.3.5.2.5.1. Use a pipette of appropriate size to collect a sub-aliquot of a sample layer of interest and transfer to an empty container.
- 11.3.5.2.5.2. If the entire sample is to be separated, pipette almost all the top layer into a new container. Draw the small volume of the top layer into the pipette along with a small portion of the second layer. Allow the two layers to separate in the pipette (like a separatory funnel). Dispense the bottom layer back into original sample container with the bulk of the bottom layer. Next dispense the remaining top layer into the container that holds the top layer.
- 11.3.5.2.5.3. Repeat as needed for each layer of interest.

11.3.5.2.6. Coliwasa

- 11.3.5.2.6.1. A coliwasa is a “coring device” designed for liquid multi-layer samples. It is a long tube with a short valve at the bottom. When designed and used properly it collects representative aliquots of each layer with each sub-aliquoting immersion.
- 11.3.5.2.6.2. The volume collected is determined by the diameter of the coliwasa and the height of the sample. The larger the tube diameter or taller the sample height, the larger the volume of sample collected.
- 11.3.5.2.6.3. Place the foot of the valve and its control rod in the multi-layer sample. Slowly slide the coliwasa tube over the control rod and close the ground glass seal of the valve at the bottom.
- 11.3.5.2.6.4. Lift the coliwasa by the control rod to keep the valve sealed.
- 11.3.5.2.6.5. Place the coliwasa over the new sample aliquot container. Grasp the top of the coliwasa tube and slowly lower the control rod a few millimeters to

open the foot valve and drain the sample into the container.

- 11.3.5.2.6.6. Repeat Sections 11.3.5.2.6.3 through 11.3.5.2.6.5 until sufficient sample aliquot has been collected.

11.3.6. Multiphase Sample Mixing Procedures

11.3.6.1. All liquid samples should be mixed by shaking unless a multiple layer subaliquoting procedure is used. This applies to emulsifiable layers such as oil and water and suspendable particulates in water. The sample must remain mixed long enough to pour out a representative aliquot. Samples that show noticeable separation in under a minute should be subaliquoted using an appropriate technique as described below.

11.3.6.2. Closed container shaking

11.3.6.2.1. The shaking process must be vigorous enough to mix and distribute analytes associated with different layers, particulates, or inside container walls.

11.3.6.3. Open container stirring

11.3.6.3.1. The sample is stirred or swirled to mix.

11.3.6.4. Wet Sediments

11.3.6.4.1. Aqueous samples for non-volatile compound analysis may contain settleable materials. If the settleable materials are to be included as part of the laboratory sample, and they will remain suspended, or can easily be re-suspended and will remain so during the subsampling operation, the sample should be handled as a liquid sample. These samples should be gently swirled for 15 seconds or slowly inverted six times to reduce heterogeneity.

11.3.6.4.2. If the liquid portion only is to be used, the settleable material must be allowed to sink to the bottom before withdrawing the subsample.

11.3.6.4.3. If the settleable material will not remain suspended and is to be included in the analysis, the sample should be treated as a multilayered sample.

11.3.7. Multi-phase Sample Subsampling Procedures

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11.3.7.1. Multi-layered samples may include liquid/liquid layers, liquid/solid samples, or solid/solid samples.

11.3.7.2. If the liquid portion of a sludge sample can be re-mixed with the solid portion and will re-separate over time, the sample should be handled as a solid sample.

11.3.7.3. If the solid portion will remain suspended or can easily be re-suspended, the sample should be treated as a liquid sample. Mixing may occur by inverting the container or with slight shaking

11.3.7.4. Layer Separating

11.3.7.4.1. Gravity/Centrifuge – The solid/liquid phase separation may be achieved by gently centrifuging the unopened container or by allowing it to sit undisturbed until the solid portion is settled.

11.3.7.4.2. Pipette – For liquid/liquid layers, separate the layers using a pipette. Each layer may be either transferred into another container or directly into the analytical vessel. Each separated portion is then handled as a homogeneous liquid sample.

11.3.7.4.3. Filter/Decant – For liquid/solid layers, the liquid subsample may be obtained by filtering or decanting the liquid portion from the solid portion. The liquid portion is then handled as a homogeneous liquid sample. The solid portion is handled as a solid laboratory sample.

11.4. Sample Analysis

11.4.1. Not applicable to this procedure

11.5. Analytical Documentation

11.5.1. Record all analytical information in LIMS, including any corrective actions or modifications to the method.

11.5.2. Record all standards and reagents in the LIMS reagents module. All standards and reagents are assigned a unique number for identification.

12. DATA ANALYSIS AND CALCULATIONS

12.1. Not applicable to this procedure.

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13. METHOD PERFORMANCE

- 13.1. The Group/Team Leader has the responsibility to ensure this procedure is performed by an associate who has been properly trained in its use and has the required experience.

14. POLLUTION PREVENTION

- 14.1. It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage, and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste management and Pollution Prevention".

15. WASTE MANAGEMENT

- 15.1. All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention".
- 15.2. Waste Streams Produced by the Method
- 15.2.1. Used wood spatulas, aluminum sheets, butcher paper; discard in solid waste.
- 15.3. Laboratory personnel assigned to perform hazardous waste disposal procedures must have a working knowledge of the established procedures and practices of TestAmerica. They must have training on the hazardous waste disposal practices upon initial assignment to these tasks, followed by annual refresher training.

16. REFERENCES

- 16.1. References
- 16.1.1. U.S. EPA, 2000. TRW Recommendations for Sampling and Analysis of Soil at Lead (Pb) Sites. EPA-540-F-00-010. OSWER 9285.7-38. April. Available on-line at: <http://www.epa.gov/superfund/programs/lead/products/sssiev.pdf>
- 16.1.2. U.S. EPA, 2003. TRW Recommendations for Performing Human Health Risk Analysis on Small Arms Shooting Ranges. OSWER 9285.7-37. March. Available on-line at: <http://www.epa.gov/superfund/programs/lead/products/firing.pdf>

- 16.1.3. U.S. EPA, 2003. Superfund Lead-Contaminated Residential Sites handbook. OSWER 9285.7-50. August. Available on-line at: <http://www.epa.gov/superfund/programs/lead/products/handbook.pdf>
- 16.1.4. Michigan DEQ SOP #213 Revision #1, Nov. 9, 2004, Soil Fractions Preparation for Lead Analysis (Creating Total, Fine and Coarse Soil Samples). Available on-line at: http://www.deq.state.mi.us/documents/deq-rrd-OpMemo_2_SoilFractionsPrepForLead.pdf
- 16.1.5. ASTM D 6323-12, Laboratory Subsampling of Media Related to Waste Management Activities, 2012
- 16.1.6. TestAmerica Canton Quality Assurance Manual (QAM), current version
- 16.1.7. TestAmerica Corporate Environmental Health and Safety Manual, CW-E-M-001, and TestAmerica Canton Facility Addendum and Contingency Plan, current version
- 16.1.8. Corporate Quality Management Plan (CQMP), current version
- 16.1.10 Revision History

Historical File:	Revision 1: 05/19/99	Revision 7: 06/10/09
	Revision 2: 02/23/04	Revision 8: 09/13/10
	Revision 3: 05/24/04	Revision 9: 09/28/11
	Revision 4: 10/13/06	
	Revision 5: 11/06/06	
	Revision 6: 05/28/08	

- 16.2. Associated SOPs and Policies, current version

16.2.1. QA Policy, QA-003

16.2.2. Glassware Washing, NC-QA-014

17. MISCELLANEOUS (TABLES, APPENDICES, ETC.)

- 17.1. Reporting limits

17.1.1. Not applicable to this procedure

- 17.2. Method deviations – None

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